SDMS Document ID
2040135

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MEMORANDUM

To: Bonnie Lavelle, Chris Weis
From: Adrian Bradley, Mary Goldade
Date: November 16, 1999
Project: Vasquez Boulevard & I-70 Site, Phase III
RE: Preparation and collection of performance evaluation samples for indoor dust sampling
cc: Project Files

This memorandum describes the methods that will be used to prepare and collect site-specific performance evaluation standards for use in the analysis of indoor dust samples collected at residences within the VBI70 site. Performance evaluation (PE) samples are used to assess the accuracy of analytical methods, and to determine if collection procedures are causing a bias on the measured concentration of target analytes in the sample matrix.

For this portion of the field sampling effort, some PE samples will be prepared using surface soil that has been collected from the yards of residences within the site boundaries. In addition, a National Institute of Standards and Technology (NIST) reference material will also be submitted to participating laboratories. NIST standards are useful for evaluating the accuracy of a laboratory's analytical procedures, but they may not be representative of the conditions that exist at a particular site. Therefore, the majority of PE samples will be prepared using soil that has been collected at this site. Four yard soil samples will be prepared.

Preparation and Certification of Indoor Dust Standards

A list of candidate yard soils that will be used as PE samples is provided in Table 1. These samples have been selected to represent the range of arsenic and lead levels expected to be present in indoor dust (approximately 60-80% of the contaminant levels in yard soils). Each soil sample should be retrieved from the archive and prepared as described below.

Contract No.: N00174-99-D-003
Delivery Order No.: 0002
Purchase Request No.: 9203.3858
EPA IAG No.: DW17953800-01-0

Standard Reference Material 2583 is a dust sample that has been prepared and certified by NIST. This sample is composed of dust collected from vacuum cleaner bags used in routine cleaning of interior dwelling spaces. This sample has been sieved to a particle size of less than 100 μm.

Sample Preparation

Each soil sample must first be oven-dried at about 100 °C, sieved to a particle size of less than 150 μm and then homogenized. The mass required for each sample must be at least 200 g after preparation. Aliquots (10 g) of each sieved material should be placed into individual bottles for subsequent use. Approximately 50-100 g of the original, unprepared soil sample must be retained in archive for future use. In the event that insufficient archived sample mass is available, please notify us and we will select an alternate for that concentration category.

Metal Concentration Screening

It is conceivable that the sieving process may cause a change in the metal concentration present in the original unsieved and resulting sieved samples. Therefore, an aliquot of the sieved fraction for each concentration category will be screened by XRF prior to its use as a PE sample. If concentration results remain within a factor of two of the values shown in Table 1, continue with sample certification. In the event that any of the samples are outside this range, contact us and we will select an alternate sample.

Sample Certification

Following drying, sieving and mixing, 4 of the10-gram aliquots of each PE sample will be sent for analysis. Samples will be delivered to two different laboratories that are also independent of the analytical laboratory for which all investigative samples will be tested. Each laboratory will measure the arsenic and lead levels in the PE samples using the same preparation and analysis methods utilized for the investigative samples. That is, each PE sample will be digested using nitric acid and tested via inductively coupled plasma spectrophotometry (ICP-trace).

The analytical results will be used to establish the nominal concentration of arsenic and lead in the dust PE samples. The nominal value is defined as the arithmetic mean of the 8 measured results. The standard deviation of the measured results will be used in development of acceptance criteria (described in the next section). Table 2 identifies the samples requiring independent laboratory certification. Certified values for the NIST dust sample are already available. Therefore, this sample will not be submitted for replicate analysis. A copy of the

certification sheet is attached (Attachment 1).

Inserting Blind Performance Evaluation Samples

Blind performance evaluation samples may be inserted into the sample train as soon as the PE samples are prepared and the approximate concentrations are verified by XRF screening. Sample certification will occur concurrently with sampling activities. In order to assess the sampling and analytical procedures separately, both vacuumed and un-vacuumed PE samples will be analyzed.

Vacuumed PE samples are prepared by placing a PE sample onto a hard surface and using the same dust collection procedure utilized at the residences at the VBI70 site. Un-vacuumed PE samples are prepared by transferring the soil directly to a sample collection bottle without vacuuming it up. If fractionation of the sample due to the vacuum process is occurring, a difference in concentration between the vacuumed and un-vacuumed PE samples will be observed.

Field sampling personnel will collect the Vacuumed PE sample using the following procedures for :

1. Weigh the initial mass (in grams) of the PE sample that will be placed onto the clean surface. Record the initial mass in the Field Logbook.
2. Measure and record the tare weight of an empty sample container in the Field Logbook.
3. Pour the entire PE sample onto a clean (lead and arsenic free) surface.
4. Collect sample using the indoor dust collection vacuum following the procedure described in the Indoor Dust Sampling SOP (SOP #ISSI-VBI70-04).
5. Remove the catch bottle from the collection vacuum and weigh the sample container containing the PE sample. Record the final weight in the Field Logbook.
6. Submit the PE sample blind to the laboratory in the labeled sample container. PE samples must be labeled in the same manner as the investigative samples to ensure anonymity.
7. Note the time, date and dust PE sample ID in the Field Logbook each time a PE sample is collected.

Field sampling personnel will collect the Un-vacuumed PE sample using the following procedures for :

1. Measure and record the tare weight of an empty sample container in the Field Logbook.
2. Pour the entire PE sample a sample container. Record the final weight in the Field Logbook.
3. Submit the PE sample blind to the laboratory in the labeled sample container. PE samples must be labeled in the same manner as the investigative samples to ensure anonymity.
4. Note the time, date and dust PE sample ID in the Field Logbook each time a PE sample is collected.

A total of three un-vacuumed and three vacuumed PE samples will be submitted for analysis for each concentration level. Table 3 provides a summary of the samples to be included in the performance evaluation.

PE Sample Data Review

Acceptance Criteria

The results of the dust PE analyses will be considered acceptable if 95% of all measured results are within the 95% confidence interval of the nominal value. That is, the acceptance criteria are defined as ± 2 times the standard deviation of the mean (nominal value) for each concentration level. If recoveries of vacuumed PE samples are outside of this acceptance criteria, the results of the un-vacuumed samples will be reviewed. If the results of the corresponding un-vacuumed PE samples are outside of the acceptance criteria, analysis of investigative samples must be discontinued, and corrective action procedures implemented until the problem is resolved.

Comparison of Vacuumed and Un-vacuumed PE Samples

In the event that a particle size fractionation process is occurring during vacuuming and a systematic concentration difference between the vacuumed and un-vacuumed PE samples is observed, a graph that compares the results of the two samples will be prepared. Linear regression analysis for each vacuumed and un-vacuumed PE sample pair will be performed for

each concentration level. This regression analysis may be used to correct dust concentrations in the vacuumed dust samples for any observed systematic bias.

Table 1. Candidate Sample for Use as Blind Standard Dust Samples

Sample ID	Category	As (ppm)[a]	Pb (ppm)[a]
3-01027-B	1	47.8	109.5
3-03421-B	2	199.6	246.0
3-00699-B	3	388.8	765.7
3-01628-B	4	595.7	455.9

a - Values shown are in the bulk soil (sieved to < 2mm particle size). Concentrations in the PE sample (sieved to < 150 μm particle size) may vary.

Table 2. Number and Types of Samples Requiring Independent Laboratory Certification

Standard Category	Laboratory 1		Laboratory 2	
	Un-vacuumed Soil Certification	Vacuumed Soil Certification	Un-vacuumed Soil Certification	Vacuumed Soil Certification
NIST	--	--	--	--
1	4	--	4	--
2	4	--	4	--
3	4	--	4	--
4	4	--	4	--

-- Not applicable. Does not require independent laboratory certification.

Table 3. Quantity of Performance Evaluation Samples Planned for Insertion During Indoor Dust Sample Collection

Standard Category	Un-vacuumed Soil Certification	Vacuumed Soil Certification
NIST	3	3
1	3	3
2	3	3
3	3	3
4	3	3

NIST - Certified Reference Material # 2583.
See Table 1 for site-specific PE standards.

Attachment 1

Contract No.: N00174-99-D-003
Delivery Order No.: 0002
Purchase Request No.: 9203.3858
EPA IAG No.: DW17953800-01-0

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2583

Trace Elements in Indoor Dust
Nominal 90 mg/kg Lead

This Standard Reference Material (SRM) is intended for use in the evaluation of methods and for the calibration of apparatus used to determine lead and other trace elements in dust. SRM 2583 is composed of dust collected from vacuum cleaner bags used in the routine cleaning of interior dwelling spaces. A unit consists of 8 g of particulate material, 99+ % of which passes a 100 µm (No. 145) sieve.

The certified values for five elements in SRM 2583 are listed in Table 1. The certified values are based on measurements using two or more independent analytical methods or a single NIST primary method. Analytical methods used for the characterization of this SRM are given in Table 2. All values are reported as mass fractions [1], on a dry basis (see Instructions for Drying), and are based on measurements using a sample mass of at least 100 mg.

Table 1. Certified Mass Fractions

Element	Mass Fraction mg/kg		
Arsenic	7.0	±	1.6
Cadmium	7.3	±	3.7
Chromium	80	±	22
Lead	85.9	±	7.2
Mercury	1.56	±	0.19

Certified Values and Uncertainties: The certified values for lead and cadmium were determined by isotope dilution mass spectrometry (IDMS). The certified values for the remaining elements were determined by combining data from two or more independent analytical methods in the manner described by Schiller and Eberhardt [2]. Because of evidence of inhomogeneity, the uncertainties for arsenic, cadmium, chromium, and lead are based on a 95 % prediction interval for the true value. This interval includes the combined effect of uncertainty components associated with material inhomogeneity, measurement uncertainty, and an allowance for differences between the analytical methods used [3]. The uncertainty for mercury, which exhibited no evidence of inhomogeneity, is based on a 95 % confidence interval for the true value, including the combined effect of uncertainty components associated with measurement uncertainty and an allowance for differences between the analytical methods used.

Expiration of Certification: The certification of this SRM lot is valid within the measurement uncertainties specified until **31 December 2010**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see Use section). However, the certification will be nullified if the SRM is contaminated or modified.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by B.S. MacDonald.

Gaithersburg, MD 20899
Certificate Issue Date: 22 June 1998*
30 Dec 96 (original certificate date)

Thomas E. Gills, Chief
Standard Reference Materials Program

*Addition of round room data and editorial revision of Table 2.

The overall direction and coordination of the technical measurements leading to the certification of this SRM were performed by J.R. DeVoe, P.A. Pella, and R.L. Watters, Jr. of the NIST Analytical Chemistry Division.

Statistical consultation was provided by S.D. Leigh and K.R. Eberhardt of the NIST Statistical Engineering Division.

Partial financial support for the development of this SRM was provided by the U.S. Environmental Protection Agency (EPA) under the direction of project managers S.L. Harper and M.E. Beard of the EPA Office of Research and Development, National Exposure Research Laboratory, Research Triangle Park, NC.

NOTICE AND WARNING TO USERS

Stability: This material is considered to be stable. NIST will monitor this material and will report any substantive changes in certification to the purchaser. Return of the attached registration card will facilitate notification.

Use: To relate analytical determinations to the certified values on this Certificate of Analysis, a minimum sample mass of 100 mg should be used and the sample should be dried according to the Instructions for Drying. Sample preparation procedures should also be designed to effect complete dissolution in order to relate the determined value to the certified value. This SRM must be stored in an air-conditioned or similar cool and dry environment away from sunlight and fumes.

Instructions for Drying: When nonvolatile elements (cadmium, chromium, and lead) are to be determined, samples should be oven dried for 2 h at 110 °C. Volatile elements (arsenic and mercury) should be determined on samples as received; separate samples should be dried according to these instructions to obtain a correction factor for moisture. Moisture corrections are then made to measurement values before comparing them to the certified values. At NIST, mass loss on drying at the time of certification was found to be 3.9 % with a standard deviation (n = 6) of 0.6 %.

COLLECTION, PREPARATION, AND ANALYSIS

Collection: The bulk of the material for this SRM was obtained from households, cleaning services, motels, and hotels from North Carolina, Maryland, Ohio, and New Jersey. The vacuum cleaner bags were collected under the direction of the Research Triangle Institute and the U.S. Environmental Protection Agency. The collection process was coordinated by E.D. Hardison and D.A. Binstock, of the Research Triangle Institute, Research Triangle Park, NC, under the leadership of W.F. Gutknecht.

Preparation: The bags were labeled to provide source identification, boxed and sent to Neutron Products, Dickerson, MD, for radiation sterilization, and then shipped to NIST for processing. The initial screening and preparation to select suitable material were directed by P.A. Pella and performed by A.F. Marlow, C. Desai, P. Seo, and D. Lillian of the NIST Analytical Chemistry Division (ACD). A sample of dust from each bag was passed through a 100 µm nylon sieve and measured by laboratory x-ray fluorescence. Only bags containing dust measuring 60 µg/g to 300 µg/g of lead were retained for preparing this SRM. The selected bags were processed by passing the contents of each bag through a coarse screen (2 mm hole size) to remove cotton and debris. Using a vibrating stainless steel sieve apparatus, the resultant material was screened in two successive steps, first through a 250 µm sieve and then a 100 µm sieve. All material passing a 100 µm sieve was combined, resieved five times through a 250 µm sieve to remove hairs, blended in cone blender and then bottled.

Analysis: Certification analyses were performed in the NIST Analytical Chemistry Division. Analytical methods used at NIST are given in Table 2.

Table 2. Methods used for the Analysis of SRM 2583*

Arsenic	**FIA-HGAAS, INAA**
Cadmium	**ID-ICPMS**, ICPMS
Chromium	**INAA, ICPMS**
Lead	**ID-ICPMS**, ID-ICPMS (CFM), XRF
Mercury	**FIA-CVAAS, INAA**

*Methods used for establishment of certified values are shown in bold-face type; methods used for information only values or to corroborate certified values are not in bold.

Methods

FIA-CVAAS	Flow injection analysis cold vapor atomic absorption spectrometry
FIA-HGAAS	Flow injection hydride generation atomic absorption spectrometry
ICP-AES	Inductively coupled plasma atomic emission spectrometry
ICPMS	Inductively coupled plasma mass spectrometry
ID-ICPMS	Isotope dilution inductively coupled plasma mass spectrometry
ID-ICPMS (CFM)	Isotope dilution inductively coupled plasma mass spectrometry with continuous flow microwave digestion [4]
INAA	Instrumental neutron activation analysis
XRF	X-ray fluorescence spectrometry (wavelength-dispersive)

NIST Analysts

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User Experience with SRM 2583

In order to demonstrate user experience with SRM 2583, a number of laboratories analyzed this material, each using its typical method. Among the participants, the range of digestion procedures used included the EPA-SW846-3051 and 3052 microwave digestion methods and the 3050A hotplate method. In addition, EPA-SW846-7470A and 7471 were used for Hg, and ultrasonic digestion methods were used for Pb. Instrumental methods included ICPMS, Graphite Furnace AAS, Flame AAS, and Hg cold vapor AAS. The results from this study were not used in calculating the certified values of SRM 2583. The results are given in Table 3. The summary statistics are based on 6 reported results for Pb, and 5 results for the other elements.

Table 3. Results of Round Robin Exercise

Element	Mean (mg/kg)	Minimum (mg/kg)	Maximum (mg/kg)	s* (mg/kg)
As	5.2	2.5	6.3	1.6
Cd	6.3	5.8	7.0	0.5
Cr	44	33	67	13
Hg	1.5	1.3	1.7	0.2
Pb	78	57	89	13

*s is one standard deviation.

REFERENCES

[1] Taylor, B.N., "Guide for the Use of the International System of Units (SI)," NIST Special Publication 811, 1995 Ed., (April 1994).

[2] Schiller, S.B. and Eberhardt, K.R., "Combining Data from Independent Chemical Analysis Methods," Spectrochimica Acta, **46B**, pp. 1607-1613, (1991).

[3] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, (1993): see also Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington DC, (1994).

[4] Beary, E.S., Paulsen, P.J., Jassie, L.B., and Fassett, J.D., "Determination of Environmental Lead Using Continuous-Flow Microwave Digestion Isotope Dilution Inductively Coupled Plasma Mass Spectrometry," Analytical Chemistry, **Vol. 69**, No. 4, (February 1997).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Telephone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet http://ts.nist.gov/srm.